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SEMI-ANNUAL PROGRESS REPORT

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X-RAY TRANSMISSION MICROSCOPE DEVELOPMENT

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X-Ray Transmission Microscope Development
Semi-annual Progress Report

1. INTRODUCTION:

Physical processes which occur at, or near, the solid-liquid interface during solidification or other phase transformations, partially determine important properties of solids. To-date, interfacial morphologies and particle-interface interactions in the respective metallic, optically opaque systems have been deduced from post-process metallographic analyses of specimens. Thus, little information is obtained about the detailed dynamics of the processes.

We are developing a high resolution x-ray microscope to view, in-situ and in real time, interfacial processes in metallic systems during freezing or even during solid-solid transformations. The X-ray Transmission Microscope (XTM) will operate in the hard x-ray range (10 to 100 keV) and achieve magnification through projection. We have obtained, using select aluminum alloys, in-situ records of the evolution of interface morphologies with characteristic lengths as small as 80 μm, interfacial solute accumulation and formation of droplets (70 μm). This was the state of the art before the ATD began in Oct. 1993. In order to improve these capabilities, we have further addressed the complex issues of resolution, contrast and minimal exposure time and are assembling an XTM with greater capability.

The anticipated capabilities of the XTM to be developed through this ATD include:

1. a resolution for specimen features of 10-100 μm,
2. solidification rates of 0.1 to 20 μm/sec,
3. temperatures up to 1100 °C with temperature gradients up to 50 °C/cm,
4. contrast sensitivities sufficient to detect 2-5 % difference in absorptance,
5. exposure times of a few seconds,
6. recording of stereo pairs for depth information.

1.1 Approach

With metallic and semiconducting samples, the penetration of macroscopic layers requires photon energies in excess of 10 keV. This precludes the use of optical approaches for imaging. Only projection radiography can be employed in this energy range of over 10 keV. Projection radiography uses the divergence of the beam from a small source. The ultimate resolution is limited by the diameter of the source. Hence, x-ray projection radiography requires micro-focus x-ray tubes. Our XTM will utilize the smallest source available. The spot size can be set down to less than one micrometer diameter.

Figure 1 schematically indicates the major components of the system and their placement. A metal sample (thickness of order mm) is contained in a specially designed, high transmittance crucible. A high temperature furnace on a translation stage imposes a temperature gradient onto the sample. The solid-liquid interface is positioned in close proximity to the focal spot of a micro-focus x-ray source. The diverging x-ray beam permeates the sample and the resulting shadow falls on an x-ray image converter. The resulting visible image is converted to a digital image by a CCD camera and stored in a computer. This image is displayed on a high resolution monitor, either in real time or after further processing (contrast enhancement, filtering, etc.). Hard copies of the images are obtained either photographically from the high resolution monitor, or from a video printer.

At typical solidification rates, motion-induced blurring limits the exposure time to a few seconds. With state-of-the-art x-ray image intensifier/camera combinations, which have a spatial resolution of order 100 μm, a magnification on the detector of some 20X is required to obtain a spatial resolution of 10 μm. Such resolution is needed to see the dendritic structures formed in solidifying metals. Since magnification is the direct ratio of detector-source to specimen-source distances, magnifications of 20X or more will require the (heated) specimen to be less than 1 cm from the housing of the x-ray tube. This leaves little room for the crucible, insulation and cooled housing creating challenging design problems for the x-ray furnace.

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Of course, such observations require sufficient contrast (difference in absorptance) between features to be resolved and the retention of this contrast by the imaging devices (x-ray converter, image intensifier, camera, recording device). In monocomponent metallic systems, contrast between solid and melt is determined by the (electron cloud) density of the two phases resulting in less than 2% radiographic (image) contrast. In alloy systems, solute segregation will lead to further contrast enhancement. The magnitude of contrast is proportional to the difference in atomic number of the components and their concentration.

1.2 Research Goals for the XTM

Research goals include studying solidification of metals and semiconductors and the dispersion of reinforcement particles in composites. Features we will observe include dendrites and cells, the effects of reinforcement particles on the morphology during solidification of Metal Matrix Composites, interfacial faceting phenomena, and solutal segregation profiles.

2. PROGRESS TO DATE:

As originally proposed, this 6 month period was for converter/detector research and development of an imaging model. No hardware was available but the x-ray source is expected to be available just after this report is submitted.

The results from the two efforts mentioned above will now be outlined in detail.

2.1 Converter/Detector Research

We have performed the initial evaluation of the converter and detector technologies based on the state of the art as it can be best determined at this time through published literature and manufacture's data. The three technologies selected are shown in the following figure.

The x-ray source was purchased with an x-ray image intensifier which is at the commercially available state of the art. The image from this intensifier will be detected using a Photometrics 200 12 bit CCD camera already available. This combination will offer the best sensitivity but will lack low energy detectability and may have poor spatial resolution and less than adequate dynamic range. The alternate technologies will be purchased for evaluation in the second year of the effort. The three candidate technologies are:

1. Conventional x-ray image intensifier (X.I.I.) coupled to cooled (visible light) CCD camera of 12 bits dynamic range. X.I.I. at best offers 10 bit dynamic range (1 part in 1000).

2. Direct conversion within the Silicon of a radiation hardened CCD designed for x-ray use. Very workable, presently only 8 bit dynamic range. Improved electronics and cooling will offer up to 14 bit dynamic range (1 part in 16000). Not so suitable for x-ray energies over 50 keV.

3. Conversion to visible light using a scintillator coating on a fiber optic face plate then capturing the image with a cooled CCD attached to the fiber optic faceplate. Technology not yet optimized, but may compete with 1 and 2 above. Potentially better signal to noise ratio than 2 above. Can go to 16 bits if a cooled visible light CCD is used which has to be protected from exposure to x-rays (using shielding and the fiberoptic faceplate).

2.1.1 X-ray Detector Research for X-ray Transmission Microscope

Evaluation of new CCD x-ray converter and camera technology is being performed. This technology is being developed at General Imaging, Florida, to use radiation hardened CCDs as a direct conversion, hard x-ray detector for medical purposes. This competitive technology may offer a superior means of seeing the features we most want to see with increasing resolution and potentially higher 'contrast' resolution properly called dynamic range.

The CCD devices provide characteristics of:

very high resolution
potentially high dynamic range (# gray levels)
high signal to noise factor
adequate sensitivity
can be placed close to specimen

Regarding the latter statement, it was determined that one could place the CCD detector closer to the specimen and still preserve a high resolution while also increasing the captured flux. At the same time, the x-ray image that passes the CCD detector can still be imaged with the intensifier. Two fields of view can be established and activities in one part of the specimen will not be lost because of the smaller field of view the CCD creates at the position closer to the specimen. Various scenarios can be considered such as using the rad-hardened CCD for digital image gathering while recording a continuous videotape (analog) sequence from the intensifier with a video camera or even a film movie camera. The possibilities multiply when one considers that the specimen to source distance determines the magnification as well as the detector to specimen distance.

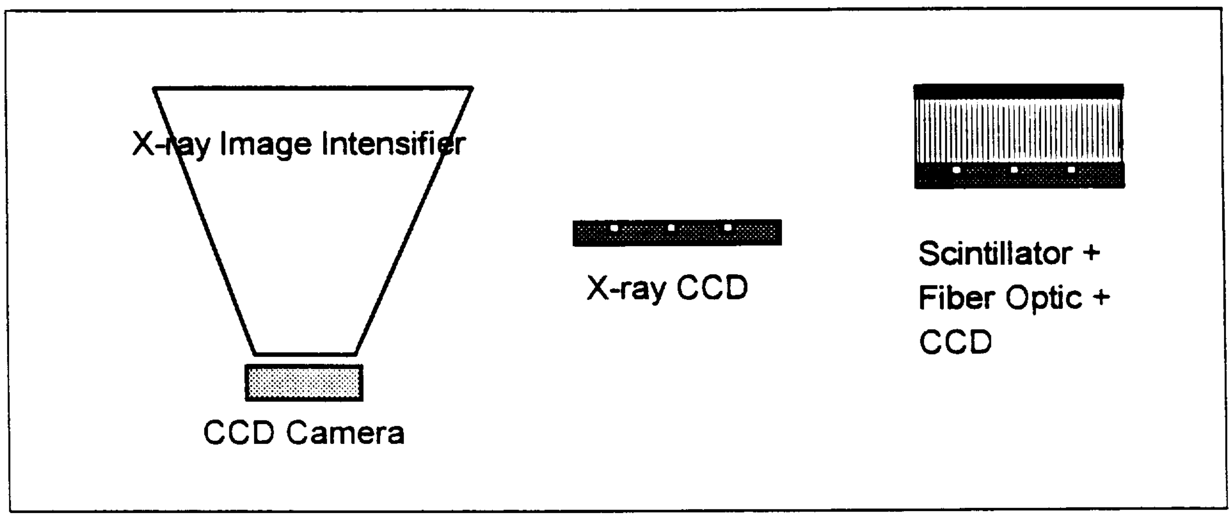


Figure 2. Schematic of the three candidate converter/detector technologies. Not to scale.
For the x-ray CCD, the detector *is* the converter.

2.1.2 Tests with Intraoral Dental X-ray Camera and Microfocus X-ray Source:

With the cooperation of Fein Focus USA, and General Imaging of Florida, we performed a performance evaluation with one of the prototype dental x-ray cameras in Atlanta. This device is commercially available for dental imaging applications. Because of its specialized use, limits to performance exist which need to be addressed with a similar unit used with the X-ray Transmission Microscope. The Fein Focus x-ray generator offered the x-ray beam conditions used during the feasibility study. The CCD device was exposed under comparable conditions to the aluminum alloy specimens during the earlier work.

Results show a high resolution (over 3X more than inherent resolution of x-ray image intensifier). In addition, of significant consequence, it was determined that the CCD was as sensitive as the x-ray image intensifier + camera combination that is presently the best performer. Due to the lack of available data from the manufacturer, such a determination would not have been possible from modeling or calculation determinations. Performing these hands-on evaluations is the most efficient way to show the proper course of action to take. It was also determined that the device was very small and easy to use. Modifications to the

CCD device and associated instrumentation should be simple enough to do by the General Imaging company and a device should be available for further evaluation in the near future. There is a potential to improve dynamic range to 14 bits by adding active cooling and better readout electronics, compared to the 10 bit limitation for best x-ray image intensifiers. This modification has not yet been done nor tested with these radiation hardened CCDs. We feel there will be no problem to make these special additions to the device since non-radiation hardened CCDs have been used this way for astronomy and spectroscopy applications. Results were obtained from a couple of resolution tests and a real specimen of 1 mm thick aluminum 3% copper alloy that was previously processed during the feasibility study but never cut for analysis. The specimen in its boron nitride crucible was radiographed by placing it onto the CCD intraoral detector and irradiating them with a 35 kVp beam to match the conditions obtained during the feasibility study. A two second exposure yielded almost identical results to the feasibility results where an intensifier and cooled CCD camera were employed. Resolution tests were performed using line pair gages which showed, with no net magnification, that the CCD intraoral detector could resolve 20 line pairs per millimeter. This is three times better than a unity magnification resolution with the x-ray image intensifier.

2.1.3 Evaluating Three Converter/detector Technologies:

Several conversion technologies have been surveyed for applicability to this project. Due to the real-time requirements, film is not suitable for use in this project except to record images of stationary specimens. Only technologies that offer instant or real-time images will be considered.

From the initial survey, three technologies have been selected for consideration. One is conventional x-ray image intensifier technology already in common use in the NDT and medical fields. Known limitations of contrast and resolution exist which indicate this technology may not be the best for this project, although, to date, it has proven to be the best when modified. The second technology only became available during the feasibility period of the effort. Since then, it has been shown to be a contender to the intensifier method. This technology uses the CCD as the x-ray photon converter and as the storage device for the converted image so it can be displayed. The third technology would be to use a cooled CCD for image collection from the visible light output from a scintillation screen which converts the x-rays to visible light. Sensitivity with this technology is the difficulty. Resolution and contrast should not be a significant problem. Such a technology is similar to the conversion process that must occur at the front end of the intensifier. Finding the best scintillation phosphor for the energy range is one part of the problem. Another is the coupling of the scintillator to the CCD to minimize losses of the converted visible light photons. Alternative scintillator technologies have been evaluated as well. The one that looked very good (until a certain raw phosphor was tried) was a fiber optic scintillation plate where the fibers in a bundle were made of scintillating glass. The light generated within the fiber would be conducted to the end of the fiber and then to the nearby CCD pixel. The value of the method is that thicker plates could be used that still preserve resolution down to the fiber diameter and spacing. However, the conversion efficiency was not as high as the crystalline phosphors at the low x-ray energies we plan to use. Scintillators were found to be better at accelerations below 50 kVp while still retaining high resolution. The best scintillator we found, which competes with CsI, is Y_2O_2S:Eu. For the 6-10 micrometer particle size, coated glass plates offered 15-16 line pairs per mm resolution.

Experience indicated improvements in the intensifier technology would be obtained when combining a cooled CCD camera with the intensifier. This would extract the most from the technology and has reached its peak. In fact, shortly after experiments with the cooled CCD, a commercial product where the intensifier and cooled CCD were integrated was marketed for the NDT industry. Due to the known abilities of the standard x-ray image intensifier and the advantages it offers for sensitivity and speed, one was purchased to be delivered with the x-ray source as standard equipment. We will be using the conventional x-ray image intensifier coupled to cooled (visible light) CCD camera of 12 bits dynamic range. The intensifier at best offers 10 bit dynamic range (1 part in 1000).

For direct conversion within the silicon (of a radiation hardened CCD) a separate device will need to be purchased for evaluation. The method is very workable but presently only an 8 bit dynamic range is available. We will try to improve the electronics and add thermoelectric cooling to perhaps offer up to 14 bit dynamic range (1 part in 16000).

The third technology employs conversion to visible light using a scintillator and/or fiber optics then capturing the image with a cooled CCD. This technology is not yet optimized, but may compete with the other two above. This is not new technology, but finding the phosphors and coupling methods between the scintillator and CCD that enhance performance make this a strong possibility. One problem is that a radiation hardened CCD needs to be used if absorbing glass is not protecting the CCD from the x-rays. Using a fiberoptic faceplate to protect the CCD from the x-rays, the further developed visible light CCDs can be employed which presently have reached 5000 x 5000 pixels in a two inch square detector. The radiation hardened detectors are only available in 770 x 576 pixel sizes now. Cooling these detectors to obtain high dynamic range is common technology. Less common is the readout electronics that extract more than 8 bits per pixels from these devices. It turns out that the use of a scintillator directly on the CCD may compromise the signal to noise ratio since so many electrons are created by the Si if an x-ray photon is captured. In addition, care must be taken to use the device in the best energy range for detection. It may be that the CCD devices are best used at low energies of less than 50 kVp acceleration. Our goal, again is to obtain 14 or more bits per pixel. Such electronics become exponentially expensive as the bit requirement is added. A 10 bit CCD system for astronomical use in the visible light range need only cost $10,000. A cooled CCD camera for astronomical use with 2000 x 2000 pixels and 12 bits of dynamic range generally costs $40,000 or more (today).

2.2 X-ray Imaging Models:

A first principles approach was used to determine the useful contrast from a sample. Using standard materials data and published x-ray spectra, the degree of absorption of the rays as they pass through the inhomogeneous sample was calculated and plotted. Resolution limits established by the converter / camera combination were introduced via MTF data for the x-ray image intensifier. The effort to date does provide useful information about the likelihood of observing certain features and, to a degree, how high a contrast the feature may have under ideal conditions.

Source flux data for 100 kVp acceleration and 50 kVp acceleration were used. For all, a tungsten target, 1 mm Al filtration, and either a 10 to 100 keV or 10 to 50 keV spectrum range were used. These are conditions available from the from Fein Focus Microfocus x-ray source being purchased.

Absorption cross-section and density data over the range 10-100 keV were obtained for Al (solid and liquid), Pb, In, B, N, Si, Cs, Au, O, S, Y, I and C elements. For compounds like SiC, SiO_2 and BN, one must first calculate mass attenuation coefficients from the elemental data and density data. The critical calculation for the absorption model comes from applying Beer's Law to obtain transmitted flux of different specimens. The following specimen configurations were used in this analysis:

1 mm thick Al (basic specimen) & 5 mm BN (crucible walls) with 1 mm Al

10 μm Pb or In particles in the 1 mm Al

100 μm SiC particles in Al

and 100 μm voids in 1 mm Al

Intensity differences (for example between Al and Al with a Pb particle) were used to obtain image contrast data as a function of photon energy from 10-100 keV. Figure 3 displays the different spectra of transmitted photons through the specimen with In and Pb particles. Contrast from the features in the specimen depends on several things. The major factors being the materials and the x-ray energy spectrum. One finds contrast varies from very little to considerable depending on the energy selected. In general, lower energies offer greater contrast. Secondly, the acceleration voltage produces x-rays from the tungsten target which have a peak output at 50% in keV photons, approximately, of the maximum acceleration voltage in kVp. This however, is more of a flux issue and therefore can be compensated with exposure times. Since the x-ray image intensifier responds to a range of photon energy (over 25 keV), the image contrast from this device may be better reflected through the use of the integrated transmission over the full range of the photon spectrum. Thus an alternative contrast from the detector would be obtained from the integration of difference of transmitted intensity vs. photon energy to obtain overall fluxes for each configuration.

The results from this model have been summarized in Table 1. Contrast of 0.01 or 1% is a goal for observation. The NDT industry uses 2% as a minimum detectable contrast.

This table shows sets of calculations for two energy ranges, up to 100 and up to 50 keV. Notice that lower acceleration can offer higher contrast as already proven with the experiments of the feasibility study. In the study, acceleration voltages of 35 kVp were selected as optimum. However, as voltage goes down, so too does the overall flux for a given current. Some experiments have shown that sufficient contrast can be obtained from higher voltages depending on the detector.

Some key features of the table show that the solid-liquid interface of Al will offer no more than 2.9% contrast if the x-ray spectrum for illumination is from a 50 kVp acceleration. It goes down to 1.4% if 100 kVp is used for the accelerating voltage. Significant contrast is obtained from heavy metal particles of either In or Pb in 1 mm Al while 100 micrometer SiC is still not likely to be seen in 1 mm of Al. Large voids are more easily seen than the SiC of the same size. The BN crucible also causes significant contrast and therefore signal losses from the BN need to be reduced to allow the best imaging conditions for low contrast microstructural details.

Modeling must continue for the detector end of the system and use manufacturer data or experimental observations to build this component of the x-ray imaging model system. Issues of resolution, contrast and sensitivity are paramount here. This is also an area that has historically been inadequately researched and needs to be addressed seriously. Another area of research and modeling that needs to be addressed is the x-ray generation itself. For the model described above, flux level was not truly addressed. Therefore exposure times and detector sensitivity evaluations cannot be made. Having stored spectra measured on other machines will not solve this problem. What will be focused upon is obtaining the capability of calculating the spectra from first principles and perhaps also the photon flux level. Two approaches are possible. One is the pure physics approach looking at atomic interactions with accelerated electrons. The other is to employ empirical criteria perhaps obtained from direct measurements from the x-ray source under various conditions. The former is not likely to be accurate and the latter is less flexible since various data sets would be needed.

Contrast vs. photon energy data was obtained from the absorption model. Then the known MTF function for the Thompson CSF X-ray Image Intensifier was applied. This is the type that is being purchased. This calculation provides a measure of the loss of contrast from an image based on feature size. If the initial contrast is low, then after conversion through the intensifier, contrast will be much lower if the features are small at the front end of the device. One distinct advantage obtained from the projection method is the ability to enlarge the features an minimize loss of contrast through the MTF problem. However, one does lose intensity and therefore longer exposure times are needed. In this project, since there is specimen motion, long exposures are a luxury we cannot afford.

It should be noted that the MTF calculation doesn't address the sensitivity of the intensifier. The intensifier only sees photons of higher energy than 25 to 30 keV. As a result, the high contrast peak of the curves occur at the energy the detector is least sensitive to. Manufacturer's data for this sensitivity variation with photon energy has been requested for a considerable time, but the data may not be available due to the difficulty of generating the x-rays in a mono-energetic form required to evaluate response as a function of energy or wavelength. The unusual difficulty in obtaining such data has slowed the progress in certain areas of the model development. Data that would appear commonplace is in fact not readily available. Further compounding the problem is the diversity of units used to describe x-ray flux. The units are derived from the measurement methods used. In medical circles, ionization gages are used and rads/hr are flux units and the energy often quoted is the dial value or acceleration voltage (kVp) which we know creates mostly 40% times this voltage of energy of photons in keV. In Physics, where CCDs and phosphors are studied, photons/cm^2/sec are used for flux units with additional specifications of wavelength or energy since radioactive x-ray sources can be used to fix the wavelength precisely.

For comparison, it is worth noting that the Non Destructive Testing (NDT) radiographic industry uses 2% minimum contrast for its detection criterion which is based on film radiography. If possible, then, we can adopt a 2% criterion and determine a resolution limit for the lower contrast from the MTF response. Unfortunately, the contrast from SiC particles in Al is so poor that we cannot expect to see the particles even when the particles are 0.1 mm in size. This coincides with the first experiments with such particles (done during the feasibility stage) where only the largest particles could be seen. These calculations warrant the effort to coat dense metallic particles with SiC for future studies. The above calculations can also be applied to predict the coated particle radiographic response.

In a similar application, the contrast of a gold deposit resolution target on a glass substrate was calculated. One company offered to make a high resolution target to measure x-ray spot focus and resolution.

To do this, a 3 micrometer deposit of gold would be ion etched to create the complex pattern. The calculations performed using the model showed a 15% contrast level would be the best obtained from such a target configuration. What is not known however, is how low the contrast might become for the smaller features due to MTF losses. Contrast loss may in fact be so high that the smaller sized features may not be visible except with magnification and long exposures on x-ray film. The model did show at least that such a target would work. The calculations also showed that better contrast would be obtained when the substrate thickness was reduced from the initial 1.5 mm down to 0.5 mm glass. These dimensions are constrained by the process used to make the target.

5. Summary:

Refinement of the models will be needed, and will be enhanced by direct measurement and experimentation with the apparatus that will arrive soon. In addition, converter and detector testing will be performed to permit modeling of their behavior as well. The models and research carried out so far have shown that some theoretical limitations do exist that make some combinations of parameters inaccessible for imaging.

4. Publications and Presentations:

Three presentations, one published paper and one abstract have been generated to date.

One presentation, by Dr. William F. Kaukler and Dr. Franz Rosenberger was at the Alabama Materials Research Conference in Sept. 1993 at A & M University in Huntsville, AL.

The second was a poster presentation by Dr. Kaukler at the Thirteenth Annual Alabama Electron Microscopy Annual Meeting held at the Huntsville Hilton in Feb. 17 and 18, 1994.

The abstract for this presentation was published in Microscopy Research and Technique vol. 28, page 452, 1994.

The third presentation was another poster presented by Dr. P. A. Curreri, Dr. W. Kaukler and Dr. F. Rosenberger at the NASA Microgravity Materials Processing Conference held at the Von Braun Civic Center in Huntsville, AL in May 24-25, 1994.

One paper, was published in August 1994 Metallurgical Transactions; and was written by Dr. William Kaukler and Dr. Franz Rosenberger entitled: X-ray Microscopic Observations of Metal Solidification Dynamics.

Table 1

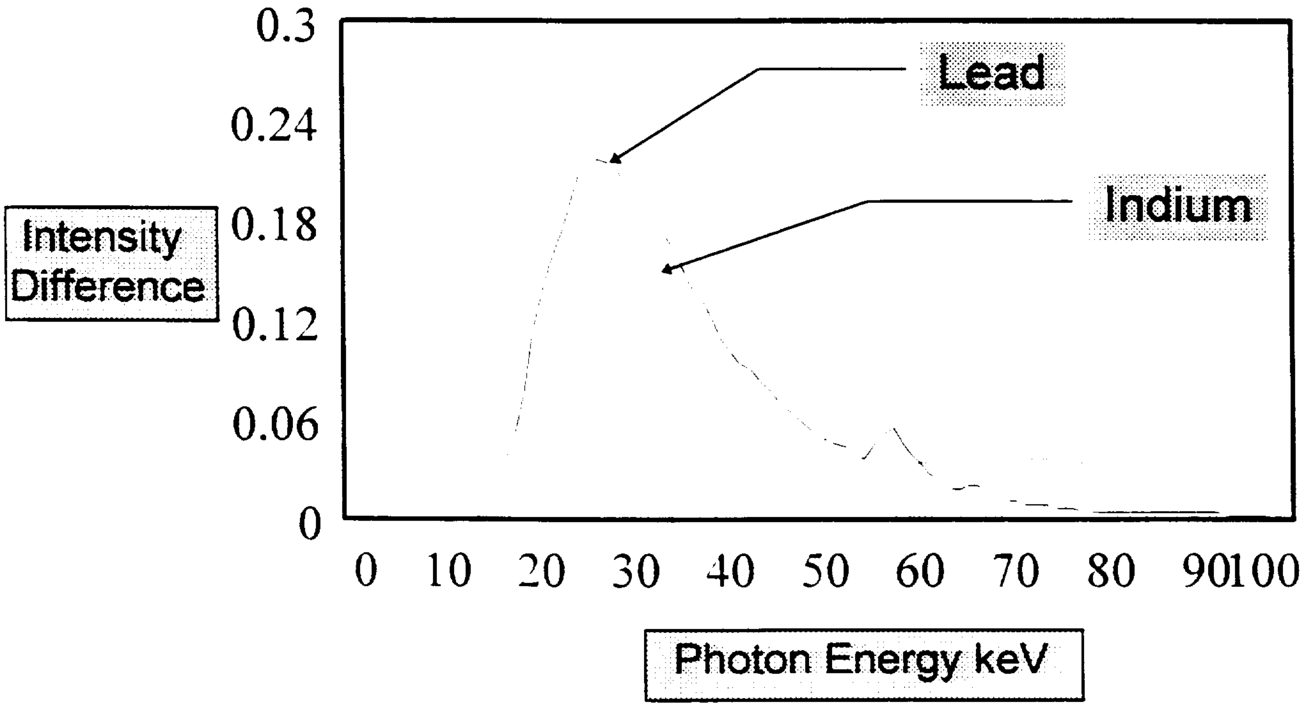
Model Results for Various Configurations

Configuration (at room temperature)	Peak Contrast (out of 1)	Loss % (10-100keV)
1 mm Al vs. nothing	0.36	19.87
1 mm solid vs 1 mm liquid Al @ melting temperature	0.02	1.42
1 mm Al + 2 mm BN vs. nothing	0.46	30.7
2 mm BN on 1 mm Al	0.12	13.52
10 μm Pb in 1mm Al	0.21	15.5
10 μm Pb in 1mm Al + 2 mm BN	0.18	14.91
10 μm Pb in 1mm Al + 2 mm BN vs. nothing	0.62	41.04
10 μm In in 1mm Al	0.17	12.73
100 μm SiC in 1mm Al	0.0027	0.231
10 μm void in 1mm Al	0.0025	-0.19
100 μm void in 1mm Al	0.027	-1.94

		(10-50keV)
1 mm Al vs. nothing	0.44	36.9
1 mm solid vs 1 mm liquid Al @ the melting temperature	0.025	2.93
2 mm BN on 1 mm Al	0.12	17.5
10 μm Pb in 1mm Al	0.26	31
10 μm In in 1mm Al	0.18	17.8
100 μm SiC in 1mm Al	0.0035	0.43
10 μm void in 1mm Al	0.0033	-0.4
100 μm void in 1mm Al	0.033	-4

INTENSITY DIFFERENCES WITH LEAD AND INDIUM PARTICLES

Difference of transmitted intensities (with and without particle) for 10 micron Lead or Indium in 1 mm Al vs. 1 mm Al



Peak values of 21% and 17% for lead and indium particles represent maximum contrast possible for the x-ray energies of 28 and 30 keV respectively. Normally, the detector sees the whole spectrum, so the integral of each peak gives a better idea of detected contrast. See Table.

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13. ABSTRACT (Maximum 200 words)

We are developing a hard X-ray Microscope for direct observation of solidification dynamics in metal alloys and metal matrix composites. The x-ray source has micrometer dimensions, producing a projected x-ray shadow giving magnified images with the resolutions required to image interior structures of the metal (if conditions produce sufficient contrast).

Detection methods, specimen preparation, and x-ray imaging conditions all play a role in creating the necessary images. Only recently have the various technologies, when taken together, permitted this undertaking. Consider that the solid-liquid interfacial contrast from 1 mm thick aluminum is only 1-3%.

Unambiguous imaging of the sample microstructure is the *tricky* part. Hard x-rays do not provide the structural contrast that softer x-rays do. Detection of the transmitted rays, however, especially for metallic systems, is easier when higher energies are used. A further difficulty is that as features become smaller, the contrast also diminishes. These issues all make detection of the microstructural features technically challenging even at the current state of the art.

This report outlines the x-ray converter and detector research and the technical attributes of the three technologies being evaluated. Secondly, results from the absorption modeling shows just how certain aluminum alloy specimens will respond to x-ray imaging.

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